

PATENT ABSTRACTS OF JAPAN

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(54) PRODUCTION OF FINE ACICULAR BOEHMITE PARTICLES

(57)Abstract:

PURPOSE: To reduce treatment temp., to accelerate the crystallization of boehmite and to reduce the cost of production by hydrolyzing a water-soluble aluminum compd. and hydrothermally treating a prepd. pseudo-boehmite sol in coexistence with hydrogen peroxide.

CONSTITUTION: One or more kinds of water-soluble aluminum compds. selected among inorg. acid salts, lower fatty acid salts, alkoxides and modified alkoxides each having a functional group substd. for part of alkoxy groups are hydrolyzed in an alkaline soln. contg. hydrogen peroxide and/or a metal peroxide and the resulting aluminum hydroxide is oxidized with oxygen generated at the time of the reaction to obtain amorphous pseudo-boehmite. Impurity ions such as Na and K ions are removed from the pseudo-boehmite by washing and then the pseudo-boehmite is dispersed in water and brought into a hydrothermal reaction in a perfectly dispersed state at 150-250° C in the presence of hydrogen peroxide in a closed system to obtain high purity crystalline acicular fine powder.

JP 06-263436 (1994)

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the manufacturing method of needlelike boehmite impalpable powder.

[0002]

[Description of the Prior Art] After adding an ammonia solution to an aluminum salt, settling gel aluminium hydroxide as a manufacturing method of boehmite and washing, drying and drying this conventionally, the method of heat-treating at 200-500 °C is adopted.

[0003]

[Problem to be solved by the invention] However, since it becomes an aluminium hydroxide cake in the state where particles condensed, in this method, it is difficult to obtain the particles of needlelike boehmite. If aluminium hydroxide is heat-treated at the temperature of 200-500 °C, in order that the diaspore of not only boehmite but the same molecular formula as it may generate simultaneously, The boehmite of the high grade was not obtained, but moreover, when heat-treated at a 450-500 °C elevated temperature, there was a problem that there was a possibility that a part of generated diaspore may change to α -Al₂O₃.

[0004] Therefore, an object of this invention is to obtain pure and needlelike boehmite impalpable powder.

[0005]

[Means for solving problem] the false boehmite which this invention hydrolyzed the aqueous aluminum compound as said The means for solving a technical problem in the alkaline solution containing hydrogen peroxide and/or an alkaline metal peroxide, and was generated - it is made to carry out water heat treatment of the sol under coexistence of hydrogen peroxide.

[0006] What is necessary is just to use at least a kind of compound chosen from the group which consists of a denaturation alkoxide replaced by other functional groups in a part of an inorganic acid salt, lower-fatty-acid salt, alkoxide, and alkoxy group as said aqueous aluminum compound. As said inorganic acid salt, an aluminium chloride, aluminum sulfate, an aluminium nitrate, etc. are mentioned, and acetate is mentioned as a typical thing of a lower-fatty-acid salt. As said alkoxide, for example aluminum(OCH₃)₃, aluminum(OC₂H₅)₃, aluminum(OC₃H_{7-n})₃, Although aluminum(OC₃H_{7-i})₃, aluminum(OC₄H₉)₃, aluminum(OC₄H_{9-i})₃, aluminum(OC₄H_{9-sec})₃, aluminum(OC₄H_{9-tert})₃, etc. are mentioned as a typical thing, It is not limited to these.

[0007] The solution of at least a kind of hydroxide and hydrogen peroxide which were chosen from the group which consists of sodium hydroxide, a potassium hydrate, ammonium hydroxide, and lithium hydroxide as an alkaline solution, Or what is necessary is just to use the solution of at least a kind of alkaline metal peroxide chosen from the group which consists of sodium peroxide, lithium peroxide, and potassium peroxide.

[0008]

[Function] In this invention, if an aqueous aluminum compound is hydrolyzed in the alkaline solution containing hydrogen peroxide or an alkaline metal peroxide, aluminium hydroxide will generate, but AlOOH , i.e., amorphous false boehmite, generates in response to the oxidation of the oxygen generated at the time of the synthetic reaction. This false boehmite has very low cohesiveness compared with aluminium hydroxide, and if water is distributed after washing this and removing impurity ion, such as Na ion, K ion, and Li ion, water heat treatment will be performed in the state where it distributed thoroughly. If this water heat treatment is performed by a closed system under existence of hydrogen peroxide, the pressure within the system of reaction will become remarkably high with the oxygen gas emitted by disassembly of hydrogen peroxide, and crystallization of boehmite will be promoted. Water heat treatment in a low temperature is conjointly made possible with this carrying out water heat treatment of the good false boehmite of dispersibility, and purity is high and it contributes to generation of crystalline needlelike boehmite particles without condensation.

[0009] It is because a heat-resistant high reaction vessel will be needed and that the temperature of water heat treatment was 150-250 °C will cause a cost rise by both sides of energy and equipment in the temperature below 150 °C, if crystallization will not fully be performed, but will be less than 90% and exceeds 250 °C.

[0010]

[Work example 1] adding an ammonia solution 28% and setting pH to nine, carrying out high-speed churning, after dissolving 0.1 mol of aluminum sulfate in 1000 ml of pure water and adding 70 ml of hydrogen peroxide solution 30% -- false boehmite -- sol was compounded. the obtained false boehmite -- after pure water washed sol 5 times, 1000 ml of pure water was distributed, and 250 ml of them was isolated preparatively, it put into the 500-ml Teflon container, and 12 ml of hydrogen peroxide solution was added to this 30%. Subsequently, this Teflon container was stored to autoclave and water heat treatment was performed at 200 °C for 8 hours. After it cooled radiationally and temperature fell to 50 °C after ending reaction, a Teflon container is taken out, ethyl alcohol performed substitution drying, the generated powder was dried [solid liquid separation was carried out using the centrifuge,] at 50 °C, and the end of dried powder was obtained.

[0011] When the obtained powder was analyzed by X-ray diffraction, it was checked that output is boehmite. When form was observed by TEM analysis to the obtained powder, the aspect ratio was a needlelike crystal of about 10 in the thickness of 35 nm, and 360 nm in length.

[0012]

[Effect of the Invention] So that clearly from the above explanation in this invention. Very little coherent amorphous false boehmite is made to generate compared with aluminium hydroxide, and it was made to carry out water heat treatment of the sol under coexistence of hydrogen peroxide by hydrolyzing an aqueous aluminum compound in the alkaline solution containing hydrogen peroxide or an alkaline metal peroxide.

Therefore, in order to be able to manufacture needlelike boehmite impalpable powder and to try to make hydrogen peroxide live together at the time of water heat treatment, without producing condensation, While the internal pressure of the system of reaction is heightened and water heat treatment is performed at a low temperature, crystallization of boehmite can be

promoted and reduction of a manufacturing cost can be attained.

Since reaction temperature is as low as 150-250 **, crystal water does not disconnect itself of boehmite structure, there is no possibility of producing the transformation to gamma-A I_2O_3 from boehmite, and the outstanding effect is acquired -- needlelike boehmite impalpable powder with high purity can be manufactured.

[Translation done.]

PRIOR ART

[Description of the Prior Art]After adding an ammonia solution to an aluminum salt, settling gel aluminium hydroxide as a manufacturing method of boehmite and washing, drying and drying this conventionally, the method of heat-treating at 200-500 ** is adopted.

[Translation done.]

TECHNICAL FIELD

[Industrial Application]This invention relates to the manufacturing method of needlelike boehmite impalpable powder.

[Translation done.]

EFFECT OF THE INVENTION

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MEANS

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[Translation done.]

OPERATION

[Function]In this invention, if an aqueous aluminum compound is hydrolyzed in the alkaline solution containing hydrogen peroxide or an alkaline metal peroxide, aluminium hydroxide will generate, but AlOOH, i.e., amorphous false boehmite, generates in response to the oxidation of the oxygen generated at the time of the synthetic reaction. This false boehmite has very low cohesiveness compared with aluminium hydroxide, and if water is distributed after washing this and removing impurity ion, such as Na ion, K ion, and Li ion, water heat treatment will be performed in the state where it distributed thoroughly. If this water heat treatment is performed by a closed system under existence of hydrogen peroxide, the pressure within the system of reaction will become remarkably high with the oxygen gas emitted by disassembly of hydrogen peroxide, and crystallization of boehmite will be promoted, Water heat treatment in a low temperature is conjointly made possible with this carrying out water heat treatment of the good false boehmite of dispersibility, and purity is high and it contributes to generation of crystalline needlelike boehmite particles without condensation.

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CLAIMS

[Claim(s)]

[Claim 1]false boehmite which hydrolyzed and generated an aqueous aluminum compound in an alkaline solution containing hydrogen peroxide and/or an alkaline metal peroxide -- a manufacturing method of needlelike boehmite impalpable powder carrying out water heat treatment of the sol under coexistence of hydrogen peroxide.

[Claim 2]A manufacturing method of needlelike boehmite impalpable powder given in Claim 1 which is solution which contains at least a kind of hydroxide in which said alkaline solution was chosen from a group which consists of sodium hydroxide, a potassium hydrate, ammonium hydroxide, and lithium hydroxide, and hydrogen peroxide.

[Claim 3]A manufacturing method of needlelike boehmite impalpable powder given in Claim 1 which is the solution of at least a kind of peroxide in which said alkaline solution was chosen from a group which consists of sodium peroxide, lithium peroxide, and potassium peroxide.

[Claim 4]A manufacturing method of Claim 1 which is a kind of compound chosen from a group which said aluminium compound becomes from a denaturation alkoxide replaced by other functional groups in a part of an inorganic acid salt, lower-fatty-acid salt, alkoxide, and alkoxy group at least, or needlelike boehmite impalpable powder given in Claim 2.

[Claim 5]A manufacturing method of needlelike boehmite impalpable powder given in any 1 of the Claims 1-4 which perform said water heat treatment at temperature within the limits of 150-250 **.

[Translation done.]